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PATENT SPECIFICATION

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(19)

(54) METHOD AND APPARATUS FOR SOLVENT EXTRACTION OF LIQUID/LIQUID MIXTURES

(71) We, DAVY POWERGAS LIMITED, of 8 Baker Street, London W1M 1DA, a British company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to solvent recovery of liquid/liquid mixtures, that is to operations of the kind whereby a liquid/liquid mixture is separated into its two components by intimately contacting the mixture with a solvent, which is not totally miscible with the mixture and in which only one of the components is soluble or in which the components are differentially soluble, to provide a two-phase dispersion, the phases are thereafter separated and then the solvent is recovered, in any convenient manner, from the appropriate separated-out phase to obtain an original component of the mixture as the residual solute.

Hereinafter, unless otherwise indicated by the context, it is assumed that only one of the component liquids is soluble in the solvent.

Known methods and apparatus for effecting solvent extraction of a liquid/liquid mixture typically involve intimately contacting the mixture and solvent in a mixing vessel to achieve mass transfer of solute and allowing the resultant dispersion to flow into a separate settling vessel wherein gravitationally controlled separation occurs, the less dense phase being retrieved from the top and the more dense phase from the bottom of the settling vessel. The output rate is not high, so that in a big scale commercial undertaking, for viability, the settling vessels require to be very large, the solvent inventory is very high and, at any one time, there is an uneconomically large hold-up of solvent and other liquids in the plant.

There have been proposed forms of apparatus designed to increase the flow rate and thus reduce the above mentioned disadvantages, but these have involved complexities which are undesirable in big scale commercial operations.

Objects of this invention are to increase the rate of throughput and thereby reduce the solvent inventory without complicating the apparatus; indeed, in some respects, the apparatus may become simplified.

It is known to use a droplet-controlling perforate packing in the operation of separating the phases of a dispersion, to enhance droplet coalescence and thereby the rate of separation-out of the dispersed phase. By a "perforate packing" is meant a body exhibiting a labyrinth formation of intersticial passages extending between an entry face and an exit face of the packing. The packing may be a labyrinth formation of tubular, ring-like, saddle-shaped and/or plate-like elements, but more conveniently it can be formed from a woven fabric or a knitted mesh fabric, in the latter case being, for instance, of the kind known as Knitmesh D.C. Packing.

By appropriately selecting the material of the packing in terms of its wettability by the dispersed phase, and the mean dimensions of the intersticial passages and its thickness, that is the distance between its inlet and exit faces, in relation to such parameters as the pressure gradient within the packing and the initial droplet dimension, it can be determined that as the dispersion passes through the packing the droplets coalesce on the surface of the packing material to form on that surface liquid films which flow through the packing to leave the exit face, by dripping action as considerably enlarged drops or even by streaming action. Such packings can be used to increase the capacity of gravity settlers by increasing the separation rate of a dispersion.

A packing has been formed throughout of single material which, in addition to being chemically inert in contact with the dispersion for obvious reasons, is selectively compatible with the dispersed phase in the sense of being wettable by that phase. For instance, to separate dispersed kerosene from water, the packing would be formed of a low surface energy material, such as polytetrafluoroethylene, whereas to separate dispersed water from kerosene the packing would be of a high surface energy material, such as stainless steel. For convenience hereinafter such packings are referred to as uniform surface energy packings.

Alternatively a packing may be formed of two different surface energy level materials of which one material is wettable by the dispersed phase but not by the continuous phase and the other material is wettable by the continuous phase but not by the dispersed phase, the packing being so constructed of two materials that throughout the packing are distributed places of contact or junctions between the different surface energy level materials it having been discovered that coalescence of the droplets is enhanced at such junctions. For convenience hereinafter, packings constructed to make use of that junction phenomenon will be referred to as "differential surface energy packings".

In accordance with the present invention, a method of separating the liquid components of a liquid/liquid mixture by solvent extraction comprises continuously feeding the mixture and a solvent for one of the component liquids to a mixing zone which is at least in part defined by a droplet coalescence enhancing perforate packing as hereinbefore defined combining the mixture and solvent by agitation in the mixing zone to form therein a dispersion, of which one phase is a solution of the soluble component liquid in the solvent, and to cause the dispersion to escape from the mixing zone through the packing to effect coalescence of the dispersed phase and separation of the phases by gravitational migration at the exit side of the packing, and thereafter recovering the soluble component liquid from the solution.

The method may be a single-stage operation, in that the proportion of solvent to liquid/liquid mixture is such that substantially all the soluble component liquid is taken up by the solvent, so that one phase of the dispersion is, or is substantially, solely the non-soluble component liquid of the mixture, and the other phase is, or is substantially, a solution of the soluble component liquid in the solvent.

Alternatively the method of the invention may be effected as a two- or more-stage operation, wherein at each stage the liquid/liquid mixture, solvent and dispersion are operated upon by the first above defined method of the invention.

In one such two- or more-stage operation only part of the total solvent required to substantially completely separate the component liquids of the mixture is introduced to the mixing zone of each stage, and from the first stage in a two-stage operation, or from each stage, except the last stage, in a more than two-stage operation, the phase comprising the non-dissolved soluble liquid component and the non-soluble liquid component is fed to the mixing zone of the next stage.

In another form of the two- or more-stage operation, the liquid/liquid mixture is introduced to the mixing zone of one end stage and the whole of the solvent is fed to the mixing zone of the other end stage, and the mixture and solvent flow in counter directions through the stages, so that in each stage only a portion of the total content of the soluble liquid component of the mixture is taken up by the solvent, the final solution is derived from the end stage at which the mixture is introduced, and the mixture depleted of the soluble component liquid is derived from the end stage at which the solvent is introduced.

The recovery of the solvent component from the solution may be a continuous operation regulated to the same rate of throughput as the phase separation, or the solution may be collected to bulk for subsequent treatment.

Also in accordance with the invention, there is provided solvent extraction apparatus comprising a mixing chamber and a separation chamber on opposite sides of a droplet coalescence enhancing "perforate packing" as hereinbefore defined, mixing or agitating means in the mixing chamber, first and second inlets for liquid to the mixing chamber, and first and second outlets for the separated phases from the separation chamber, whereby, in use of the apparatus for separating the liquid components of a liquid/liquid mixture by solvent extraction, a solvent and a liquid/liquid mixture may be continuously fed to the mixing chamber by means of the first and second inlets respectively, the mixing or agitating means serving to form a dispersion and to effect mass transfer in the mixing chamber and further serving to cause the dispersion to escape therefrom through the packing.

The packing may be a uniform surface energy packing, but preferably will be a differential surface energy packing.

The mixing chamber may be an inner chamber within and separated by the per-

forate packing from the separating chamber.

The mixing chamber can be cylindrical, being ordinarily, but not necessarily, adapted for use with its axis vertical.

In another arrangement the mixing chamber and separating chamber are in end-to-end relation on opposite sides of the packing.

The invention is further described with reference by way of example to the diagrammatic drawings which accompanied the Provisional Specification, wherein:—

Figure 1 represents a form of apparatus including a cylindrical packing;

Figure 2 represents an end-to-end arrangement of the chambers;

Figure 3 represents a multi-stage form of the apparatus; and,

Figure 4 represents another multi-stage form of the apparatus.

Referring to Figure 1, a housing comprising a cylindrical outer wall 1, an upper end plate 2 and a lower end plate 3, is divided by a cylindrical packing 4 of knitted mesh construction into a central mixing chamber 5 and an annular separating chamber 6. In the mixing chamber 5 is a mechanical agitator comprising several rotary impellers 7 carried by an axial shaft 8 which can be driven by any convenient means outside the housing. Upper and lower inlet pipes 9 and 10 respectively provide for the liquid/liquid mixture and a solvent to be fed separately into the mixing chamber 5, the heavier fluid through the inlet 9 to the top of the chamber and the lighter fluid through the inlet 10 to the bottom of the chamber. From the separating chamber 6, through the lower end plate 3, extend a lower outlet pipe 11 from the lower part of the chamber and an upper outlet pipe 13 from the upper part of the chamber, the inner ends of the pipes 11, 13 being protected by baffles 12 and 14 respectively.

The purpose of the agitator 7 is to effect vigorous mixing together of the liquid/liquid mixture and solvent as fed into the mixing chamber 5 to create by mass transfer a dispersion of which, in a single-stage operation, one phase is primarily a solution of one component liquid of the mixture in the solvent and the other phase is primarily the other component liquid of the mixture. There can be used any convenient form of agitator which will have the required effect. It is not necessarily a mechanical agitator; for instance the mixing may be effected by interference streaming and/or static mixing, by so forming the inlets that the solvent and/or mixture, pumped to the mixing chamber are introduced through specially directed and/or shaped orifices which immediately bring the fluids together intimately and forcibly promote the required contact of the solvent with the soluble component liquid and the disturbance which assists the formation of the dispersion.

The action of the agitator 7 in the mixing chamber 5 generates substantial kinetic energy in the dispersion, acting in the sense to drive the dispersion through the packing 4 from the chamber 5. In order for the subsequent action in the separating chamber 6 to proceed in the desired manner it would be undesirable for the dispersion to enter that chamber in a state of agitation. Also the effect of the kinetic energy of the droplets in the dispersion will be to tend to prevent coalescence occurring. It is a significant aspect of this invention that although the packing 4 is perforate, as above described, and although it is the only barrier between the mixing and the separating chambers, the nature of its structure as a labyrinth formation of intersticial passages extending between its inner entry face and its outer exit face provides that, by appropriately selecting its thickness, the kinetic energy is substantially entirely absorbed within the packing, causing to be developed therein an effective pressure gradient to assist the movement of the dispersion through the packing without it emerging violently at the exit face. In the passage of the dispersion through the packing 4 the absorption of kinetic energy results in the establishment of the condition which allows coalescence of the dispersed phase to occur and the coalescence is enhanced in the manner, already described, which characteristic of the packing. At the exit face of the packing 4, coalescence will have proceeded to such a degree that the coalesced liquid will issue as running streams at that face, and final separation of the phases can proceed rapidly in the separating chamber 6. In the separating chamber 6, the coalescence being substantially complete, separation occurs continuously and at a high rate, the heavier phase separating out to the bottom of the chamber 6 and the lighter liquid phase to the top of the chamber, to flow out through respectively the lower outlet pipe 11 and the upper outlet pipe 13. The baffles 12, 14 are designed as additional safe-guards to ensure that only liquid of the appropriate phase will escape at each outlet. Thereafter solvent recovery to obtain the dissolved liquid component can be carried out in any desired manner.

The described arrangement has the special advantage that, as compared with the use of a necessarily large settling vessel for affecting within one vessel coalescence of a dispersed phase and separation of the phases as part of the operation of solvent

extraction of a liquid/liquid mixture, only a small separating chamber is required because coalescence is substantially completed at a higher rate. Thus the solvent inventory, or hold up, can be considerably reduced and the operative capacity per unit volume of the equipment increased. It is also significant that although the packing, by its inherent nature, is permeable, in that fluid will pass through it from the mixing chamber to the separating chamber as coalescence proceeds within the packing it also has the special property of absorbing the kinetic energy generated in the mixing chamber, in which sense it serves effectively to contain the mixing zone, so that only the packing itself is required to separate the two chamber.

If the natures of the liquid components and solvent are such that the ambivalent range is narrow, i.e. the range of variation of the composition of the dispersion in which phase inversion may occur is small, then, if the composition is not within that range, the packing 4 can be a uniform surface energy packing as above described. It will however be appreciated that if phase inversion occurs such a packing will not then function to enhance the coalescence of the dispersed phase. Unless it can be determined that the conditions are such that phase inversion will not occur, and/or if the ambivalence range is not appropriately narrow, it is preferred to use a differential surface energy packing as above described, which will function irrespective of which is the dispersed phase in the event of phase inversion or of perhaps the dispersion being in a critical condition within the ambivalence range.

It can also be established that if contacting of the liquid/liquid mixture and solvent has not been completed in the mixing chamber 5, e.g. to the extent that the solvent action is complete, the action can also continue in the packing itself.

Figure 2 illustrates a mixing chamber 15 and separating chamber 16 in end-to-end arrangement on opposite sides of the packing 17, the arrangement being simply formed by mounting the packing 17 in the form of a transverse partition in a housing 18. Inlet pipes 19, 20 provide for the feeding of a liquid/liquid mixture and a solvent to the mixing chamber 15, and outlet pipes 21, 23 associated with baffles 22, 24 provide for the withdrawal of the separated phases from the separating chamber 16. Again the mixing chamber 13 is provided with a mechanical mixer 25. The manner of operation and possible modifications of this arrangement are substantially the same as described with reference to Fig. 1.

Figure 3 illustrates an arrangement for carrying out the method of the invention as a parallel multi-stage operation. The operation involves N stages and the apparatus comprises N mixer/separator units *1* to *n*, each of which can be one of the devices described above. The first unit *1* has an entry 26 to its mixing chamber for the liquid/liquid mixture and each unit has solvent entry 27. Each unit also has an outlet 28, from its separating chamber, for one separated-out phase which is here assumed to be the solution all the outlets 28—28 leading to a common main outlet 29. Adjacent units of all except the last unit *n* have a direct connection 30 between the outlet for the other phase from the separating chamber of the upstream unit to the other entry to the mixing chamber of the downstream unit. The other outlet 31 from the separating chamber of the final unit *n* will lead to any convenient collecting device.

In this arrangement the total inflow of solvent is divided among the several stages so that only a portion thereof is introduced at the solvent entry 27 of each unit. In the first unit *1* only part of the soluble component liquid of the mixture fed in at the mixture entry 26 will be dissolved, the solution will pass out through its solution outlet 28 to the common main outlet 29, and a mixture of the insoluble component liquid and a reduced proportion of the soluble component liquid will pass through the interconnection 30 to the next unt *2*. In that next stage *2*, with more solvent introduced, the operation will be repeated so that more solution will pass therefrom to the main outlet 29 and the proportion of soluble component liquid passing to the next stage *3*, through the interconnection 30, will be further decreased. The total amount of solvent and the rate of feeding it to the several stages can be so regulated that in the final stage *n* the solution of the soluble component liquid is completed and only the non-soluble component liquid of the original mixture issues at the final outlet 31.

Figure 4 represents apparatus for carrying out the method of the invention as a counter-current cascade multi-stage operation. The illustrated arrangement, for effecting a four-stage operation, comprises four mixer/separator units 1—4, each of which can be one of the devices described with reference to Figures 1 and 2. The four units consist of a left-hand end unit 1, two intermediate units 2,3, and a right-hand end unit 4. The arrangement provides for the liquid/liquid mixture to be fed in at an inlet 32 to the mixing chamber of the left-hand end unit 1 and for all the solvent to be fed in at an inlet 33 to the mixing chamber of the right-hand end unit 4. Each pair of adjacent units are interconnected by a connection 34, providing for outflow from the

separating chamber of the left-hand unit to the mixing chamber of the right-hand unit, and a connection 35, providing for inflow to the mixing chamber of the left-hand unit from the separating chamber of the right-hand unit. The arrangement also provides for the final solution of the soluble component liquid of the liquid/liquid mixture to be withdrawn at an outlet 37 from the separating chamber of the left-hand end unit 1 and for the non-soluble component liquid of the mixture to be obtained at an outlet from the separating chamber of the right-hand end unit 4.

It will be seen that there are two oppositely directed flow streams through the cascade of units, one directed towards the right-hand from the liquid/liquid mixture inlet 32, through the units and interconnections 1, 34, 2, 34, 3, 34, 4 to the outlet 36 for the non-soluble component liquid of the mixture, and the other directed towards the left-hand from the solvent entry 33, the units and interconnections 4, 35, 3, 35, 2, 35, 1 to the outlet 37 for the solution of the soluble component liquid of the mixture.

When this multi-stage operation is in progress, the fluids to be mixed by agitation in the left-hand end unit 1 are the initial liquid/liquid mixture and a non-saturated solution of the soluble component liquid which has been taken up by the solvent in its progress through the other units 4, 3, 2, so that in the left-hand end unit 1 some soluble component liquid will be taken up from the initial mixture by that non-saturated solution. Conversely, in the right-hand end unit 4 the fluids to be mixed by agitation are the initial solvent and the remnant of the liquid/liquid mixture from which, in its passage through the other units 1, 2, 3 almost all the soluble component liquid has been removed, so that, in the right-hand end unit 4, substantially all the remaining soluble component liquid will be taken up by the solvent, leaving substantially only the non-soluble component liquid to flow from the outlet 36. In the right-hand direction of flow through successive units the proportion of soluble component liquid in the mixture will progressively decrease as, in the left-hand direction of flow, the amount of soluble component liquid taken into solution progressively increases. It may be that an acceptable result can be obtained by using less than four units, or in order to ensure a very high degree of efficiency, it may be desirable to use more than four units.

The multi-stage operations tend to maximise the efficiency of the improved method of liquid/liquid mixture separation by solvent extraction in that the throughput rate can be high, the solvent inventory can be low and, because the vessels can be of smaller dimensions than hitherto, the hold-up of liquid at any time can be small enough to be economically attractive. Where circumstances permit, similar advantages can be achieved by carrying out the method as a single-stage operation. Significantly, the knitted mesh packing has the advantage that not only does it speed up the process due to its ability to accelerate coalescence and thereby separation-out of a dispersed phase and, in the preferred case of a differential surface energy packing, to be unaffected by phase inversion, but also will serve by itself as an effective partition between a mixing chamber and a separating chamber.

It can be assumed that in the described method the dispersion will be substantially a primary dispersion, that is the dispersed phase will have a droplet size of means diameter greater than 100 microns. It is however anticipated that circumstances may arise in which there may be present droplets of smaller dimensions, as in secondary dispersions or hazes. It has been established that in some circumstances the separation of an enlarged droplet from a coalescence enhancing packing may be accompanied by the formation of a smaller droplet of secondary dispersion dimensions. If in the method of liquid/liquid mixture separation by solvent extraction an undesirable proportion of secondary dispersion dimension droplets may occur, the apparatus can be adapted to include in combination with the knitted mesh packing another form of packing which has the function of coalescing such small droplets to at least primary dispersion dimensions.

In the description with reference to the drawings it has been assumed that only one component liquid of a liquid/liquid mixture is soluble in the solvent. However there may be circumstances in which the component liquids are differentially soluble so that the two phases of the dispersion are two separate solutions. In that case the separate liquids of the original mixture will eventually be derived by separately treating the resultant solutions. It will be appreciated that where only one of the component liquids is soluble in the solvent, the resultant solution may not necessarily be the dispersed phase. Also, although the reference to a solution suggests that a dissolved component of a liquid/liquid mixture may be recovered by merely driving off the solvent, there may be circumstances in which although the component liquids are separated by the use of a solvent, the solution may subsequently be treated otherwise than by merely driving off the solvent, e.g. to obtain an end product which is not

precisely the original component liquid of the mixture.

WHAT WE CLAIM IS:—

1. A method of separating the liquid components of a liquid/liquid mixture by solvent extraction, comprising continuously feeding the mixture and a solvent for one of the component liquids to a mixing zone which is at least in part defined by a droplet coalescence enhancing "perforate packing" as hereinbefore defined combining the mixture and solvent by agitation in the mixing zone to form therein a dispersion, of which one phase is a solution of the soluble component liquid in the solvent, and to cause the dispersion to escape from the mixing zone through the packing to effect coalescence of the dispersed phase and separation of the phases by gravitational migration at the exit side of the packing and thereafter recovering the soluble component liquid from the solution.

2. A method as claimed in Claim 1, wherein substantially all the soluble component liquid is taken up by the solvent.

3. A method as claimed in Claim 1, wherein some of the soluble component liquid of the mixture is taken up by the solvent and, after separation of the phases of the dispersion, the mixture containing a reduced proportion of the soluble component liquid is subjected to a further stage of treatment by the method claimed in Claim 1.

4. A method as claimed in Claim 1, wherein some of the soluble component liquid of the mixture is taken up by the solvent and, after separation of the phases of the dispersion, the mixture containing a reduced proportion of the soluble component liquid is subjected to further treatment by the method claimed in Claim 1 in each of two or more successive stages, the liquid/liquid mixture introduced to the mixing zone of each of said two or more stages being a phase separated from the dispersion in the preceding stage.

5. A method as claimed in Claim 3 or Claim 4 for separating the component liquids of a liquid/liquid mixture, in which the sum of the rates of inflow of solvent at the several stages is not less than is required to take up all the soluble component liquid of the liquid/liquid mixture.

6. A two-stage method of separating the component liquids of a liquid/liquid mixture by solvent extraction, comprising two successive stages of treatment by the method claimed in Claim 1, wherein the liquid/liquid mixture is introduced to the mixing zone of the first stage and the solvent is introduced to the mixing zone of the second stage, an output from the first stage of the liquid/liquid mixture with reduced content of the soluble component liquid is introduced to the mixing zone of the second stage and an output from the second stage of a non-saturated solution of the soluble component liquid is introduced to the mixing zone of the first stage, the mixture depleted of the soluble component liquid is derived as the output from the second stage and the solution to be treated to recover therefrom the soluble component liquid is derived as an output from the first stage.

7. A multistage method of separating the component liquids of a liquid/liquid mixture by solvent extraction, comprising more than two successive stages of treatment by the method claimed in Claim 1, wherein the mixture is introduced to the mixing zone of, and the solution of the soluble component liquid is derived from, a left-hand end stage, the solvent is introduced to the mixing zone of, and the mixture depleted of the soluble component liquid is derived from, a right-hand end stage, the flow directions through the end stages and intermediate stages are towards the left-hand for the solvent and towards the right-hand for the mixture, and the connections between ths stages of each pair of adjacent stages provide for the soluble component liquid content of the mixture to become progressively reduced and the soluble component liquid content of the solution to become progressively increased.

8. Solvent extraction apparatus comprising a mixing chamber and a separation chamber on opposite sides of a droplet coalescence enhancing "perforate packing" as hereinbefore defined, mixing or agitating means in the mixing chamber, first and second inlets for liquid to the mixing chamber, and first and second outlets for the separated phases from the separation chamber, whereby, in use of the apparatus for separating the liquid components of a liquid/liquid mixture by solvent extraction, a solvent and a liquid/liquid mixture may be continuously fed to the mixing chamber by means of the first and second inlets respectively, the mixing or agitating means serving to form a dispersion and to effect mass transfer in the mixing chamber and further serving to cause the dispersion to escape therefrom through the packing.

9. Apparatus as claimed in Claim 8, wherein the packing is a "differential surface energy packing" as hereinbefore defined.

10. Apparatus as claimed in Claim 8 or Claim 9, wherein the mixing chamber is an inner chamber within, and separated by packing from, the separating chamber.

11. Apparatus as claimed in any one of

Claims 8 to 10, wherein the mixing chamber is cylindrical.

12. Apparatus as claimed in Claim 8 or Claim 9, wherein the mixing chamber and separating chamber are in end-to-end relation on opposite sides of the packing.

13. A method of separating the component liquids of a liquid/liquid mixture, substantially as herein described with reference to any one of Figures 1 to 4 of the drawings accompanying the provisional specification.

14. Solvent extraction apparatus substantially as herein described with reference to Figure 1 or Figure 2 of the drawings accompanying the provisional specification.

15. Solvent extraction apparatus substantially as herein described with reference to Figure 3 of the drawings accompanying the provisional specification.

16. Solvent extraction apparatus substantially as herein described with reference to Figure 4 of the drawings accompanying the provisional specification.

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1443704 PROVISIONAL SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale.*

Sheet 1

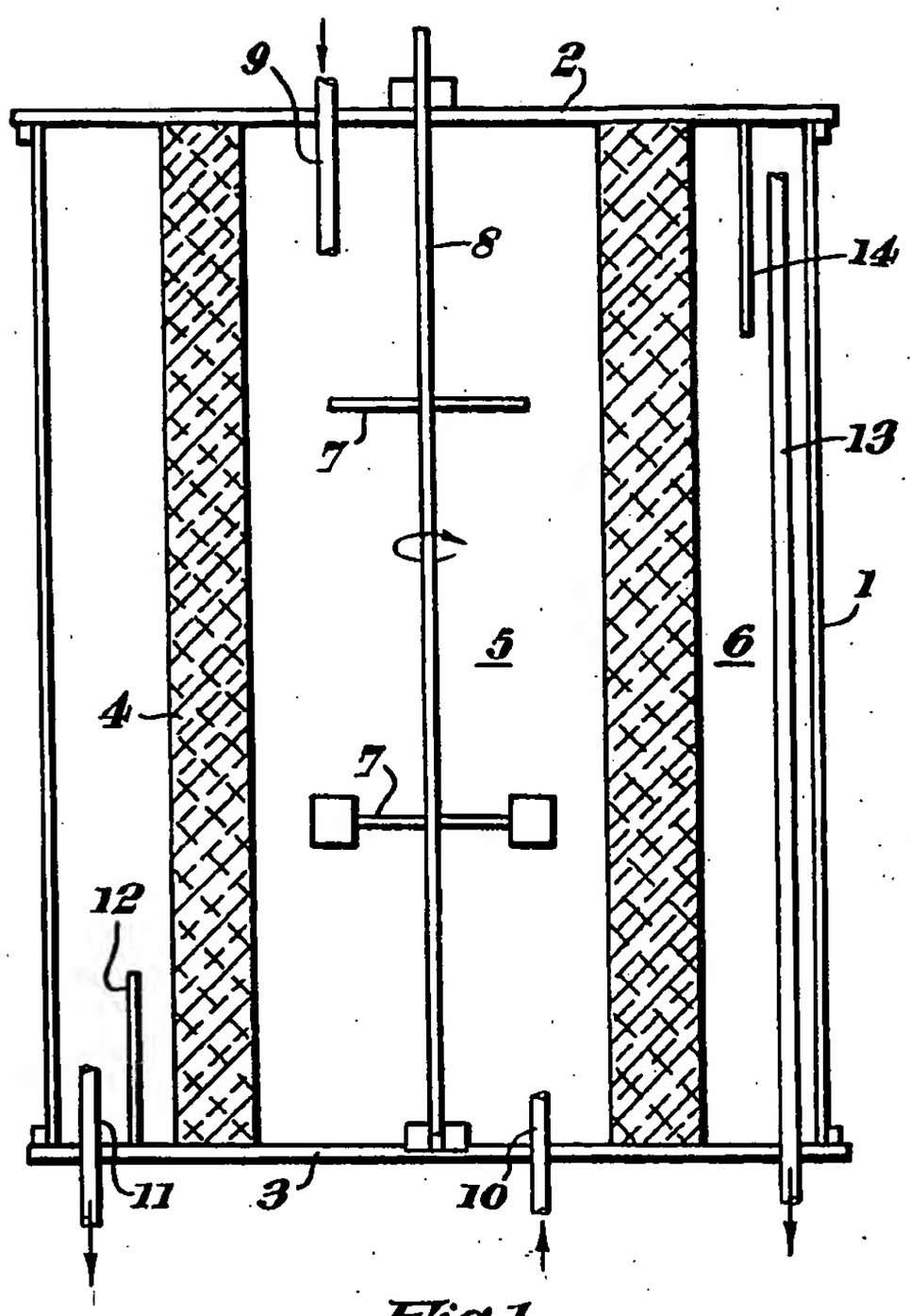

Fig. 1.

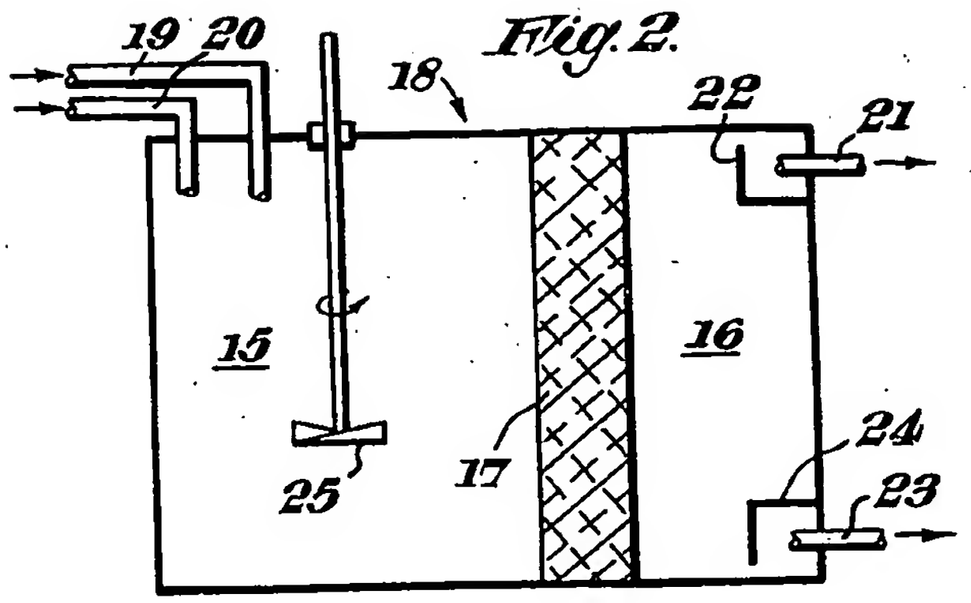

Fig. 2.

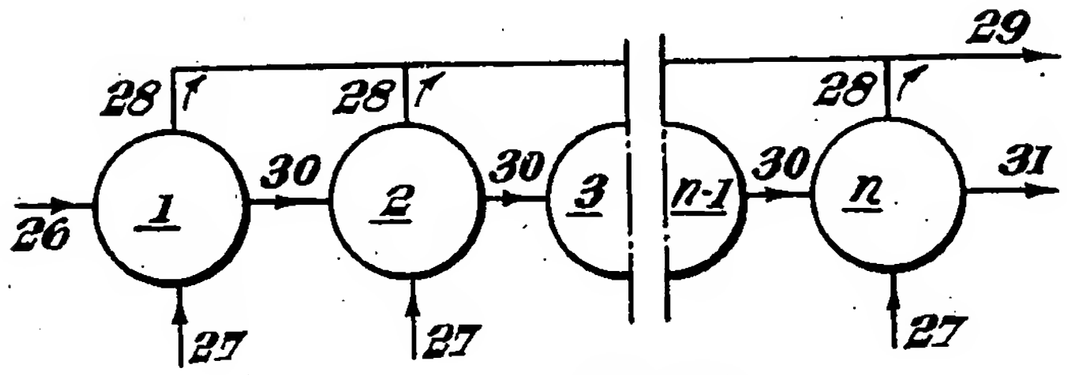

Fig. 3.

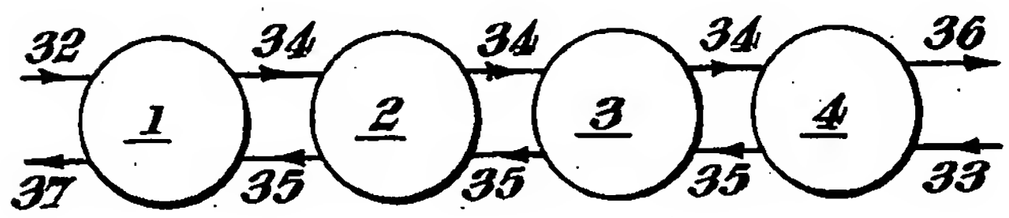

Fig. 4.